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## Description

The invention relates to a biaxially stretch-oriented, white film having a supporting layer based on propylene polymers, which contains a mixture of particles of calcium carbonate and titanium dioxide and has at least one outer layer on one or both surfaces.

A film of this type is known for example from EP-A 0 244 614. The supporting layer of this film consists of a polymer mixture of polypropylene and polystyrene and contains 5 to 15% by weight of powdery fillers. As suitable fillers, mixtures of calcium carbonate, silicon dioxide, and titanium dioxide are also used. The particle size is in the range of 2 to 5 micrometers. It is also the state of the art to incorporate titanium dioxide pigments with a coating of silicic acid and alumina into thermoplastic materials (DE-A 27 40 561). EP-B 0 044 515 teaches to use  $\text{TiO}_2$  pigments coated with the oxides of aluminum, zinc, titanium, zirconium, and/or magnesium as well as with an organic substance such as long-chain fatty acids, alcohols, or polydimethylsiloxane in plastic coatings, e.g. of polyethylene, for paper. Finally it is known from DE-A 26 46 965 to use  $\text{TiO}_2$ - or  $\text{CaCO}_3$  particles with a coating of a fatty acid ester as a filler in thermoplastic synthetic resins, e.g. polypropylene. A polypropylene film was furthermore described that contains  $\text{TiO}_2$  particles coated with  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , which are optionally coated with a polymer organic silicon compound or a polyol (DE-A 38 17 909).

The object of the present invention is to improve the degree of whiteness and the opacity of films of the initially named type. The film should in addition have good aging properties. This is understood to mean the property that the polymers under the influence of electromagnetic radiation such as light and in particular UV rays show only slight degradation phenomena or embrittlement, which lead to an impairment of the mechanical properties and yellowing of the white film. In particular the tendency to yellowing causes problems when the white film is used as an effective advertising wrapping of objects, e.g. as a packaging film, label, or as a laminating film on cardboard, paperboard, or paper. Furthermore the film should form a good barrier against light and in particular UV radiation, so that it is particularly suitable for the packaging of radiation-sensitive goods such as e.g. fatty foods. A further object of the invention is to indicate a cost-effective process for the production of this film.

This object is achieved by the film with the features named in Claim 1 as well as by the process according to Claim 15. The dependent claims indicate suitable developments of the film or the process. The propylene polymer of the supporting or base layer of the film is usually an isotactic homopolymer or copolymer of propylene with ethylene or alpha-olefins having 4 to 8 C atoms or a mixture of propylene homopolymers and propylene copolymers and/or other polyolefins, in particular having 2 to 6 C atoms, whereby the mixture contains at least 50% by weight, in particular at least 75% by weight, of propylene homopolymer. It is advisable for the isotactic homopolymer to have an n-heptane-soluble proportion of max. 15% by weight. In the

copolymers, the comonomer amount of ethylene and the alpha-olefins having 4 to 8 C atoms is generally max. 10% by weight, relative to the copolymer. Preferred comonomers are ethylene and 1-butene. Suitable polyolefins in the polymer mixture are HDPE, LDPE, and LLDPE, whereby the proportion of these polyolefins respectively does not exceed 15% by weight, relative to the polymer mixture. It is advisable for these polymers of the base layer to have a melt flow index in the range of 0.5 g/10 min to 8 g/10 min at 230°C and 2.16 kilogram-force load (DIN 53 735), in particular of 1.5 g/10 min to 4 g/10 min.

The supporting layer contains a mixture of particles of titanium dioxide and calcium carbonate. The titanium dioxide particles consist of anatase or rutile, preferably predominantly rutile, which has a higher covering power than anatase. In a preferred form of embodiment the titanium dioxide particles consist at least 95% by weight of rutile. They can be produced by a customary process, e.g. by the chloride or the sulfate process. Their proportion is 1.5 to 5% by weight, relative to the supporting layer; the mean particle size is relatively small and is preferably 0.15 to 0.30 micrometers.

The titanium dioxide particles have a coating of inorganic oxides like that customarily used as a coating for  $\text{TiO}_2$  white pigment in papers or paints to improve their resistance to light. As is known,  $\text{TiO}_2$  is photoactive. Under the influence of UV rays, free radicals form on the surface of the particles. These free radicals can migrate to the film-forming constituents of the coating material, which leads to degradation reactions and yellowing. The particularly suitable oxides include the oxides of aluminum, silicon, zinc, or magnesium, or mixtures of two or more of these compounds.  $\text{TiO}_2$  particles with a coating of several of these compounds are described e.g. in EP-A 0 044 515 and EP-A 0 078 633. Moreover the coating contains organic compounds having polar and non-polar groups. During production of the film by extrusion of the polymer melt, the organic compounds must be adequately thermostable. Polar groups are for example -OH, -OR, -COOX, (X = R, H or Na, R = alkyl having 1 to 34 C atoms). Preferred organic compounds are alkanols and fatty acids having 8 to 30 C atoms in the alkyl group, in particular fatty acids and primary n-alkanols having 12 to 24 C atoms, as well as polydiorganosiloxanes and/or polyorganohydrogensiloxanes such as e.g. polydimethylsiloxane and polymethylhydrogensiloxane.

The coating on the titanium dioxide particles usually consists of 1 to 12 g, in particular 2 to 6 g, of inorganic oxides and 0.5 to 3 g, in particular 0.7 to 1.5 g, of organic compound, relative to 100 g of titanium dioxide particles. The coating is applied onto the particles in aqueous suspension. The inorganic oxides are precipitated in the aqueous suspension from water-soluble compounds, e.g. alkali-, in particular sodium, aluminate, aluminum hydroxide, aluminum sulfate, aluminum nitrate, sodium silicate (water glass), or silicic acid.

Inorganic oxides such as  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  are understood to mean also the hydroxides or their various dehydration stages such as e.g. hydrated oxides, without knowing their exact composition and structure. After calcination and grinding in an

aqueous suspension, the hydrated oxides, e.g. of aluminum and/or silicon, are precipitated onto the  $\text{TiO}_2$  pigment; the pigments are then washed and dried. This precipitation can thus take place directly in a suspension, as occurs in the production process after the calcination and the subsequent wet grinding. The precipitation of the oxides and/or hydrated oxides of the respective metals takes place from the water-soluble metal salts in the known pH range; for aluminum, for example, aluminum sulfate in aqueous solution (pH lower than 4) is used, and the hydrated oxide is precipitated by adding aqueous ammonia solution or sodium hydroxide solution in the pH range between 5 and 9, preferably between 7 and 8.5. If a water glass or alkali aluminate solution is used as the starting point, the pH of the preliminary  $\text{TiO}_2$  suspension should be in the strongly alkaline range (pH higher than 8). The precipitation then takes place by adding mineral acid such as sulfuric acid in the pH range of 5 to 8. After the precipitation of the metal oxides, the suspension is stirred for a further 15 min to about 2 h, whereby the precipitated layers experience an aging. The coated product is separated from the aqueous dispersion and after washing at elevated temperature, in particular at 70 to 110°C, is dried.

The absolute particle size of titanium dioxide and calcium carbonate should not exceed 10 micrometers, since larger particles impair the optical properties of the film as well as the processing safety during the production of the film. Compared with the titanium dioxide particles, the calcium carbonate particles are usually used with a larger average diameter, of 0.7 to 3.0 micrometers, in particular 0.8 to 1.2 micrometers. It is advisable for the proportion of calcium carbonate particles to be 4 to 6% by weight, relative to the supporting layer, whereby a total amount of titanium dioxide- and calcium carbonate particles of 10% by weight, relative to the supporting layer, is adequate for a high degree of whiteness and high opacity.

The calcium carbonate particles consist e.g. of natural powdered rock, e.g. of calc-spar, limestone, calcite, or chalk, in so far as they are not too strongly contaminated by other substances. Carbonate particles produced by precipitation can also be used.

Precipitated calcium carbonate can be produced by various methods. Usually natural powdered rock based on calcium carbonate, in particular natural limestone, is decomposed into calcium oxide and carbon dioxide above 900°C for this purpose. After the burnt lime has been quenched with water, it is carbonated with purified carbon dioxide. The calcium carbonate particles are obtained in aqueous suspension.

The calcium carbonate particles preferably have a coating based on hydrophobic compounds, in particular compounds containing hydrocarbon chains that have at least 8, preferably at least 12, in particular at least 14, C atoms. Particularly preferred are saturated organic fatty acids having 8 to 30, in particular 14 to 24, C atoms, their sodium- or potassium salts or their alkyl esters, in particular having 1 to 18 C atoms, as well as natural or synthetic waxes based on hydrocarbons, acids, esters, soaps, or acid amides, polypropylene waxes, or polyethylene waxes. The fatty acids, their salts and esters, are obtained in particular from animal or plant fats or oils, e.g. from soybean oil, palm oil,

coconut oil, or peanut oil, and as a result are present as a mixture of different higher fatty acids. It is particularly preferred to use palmitic acid, stearic acid, and hydroxystearic acid, their alkali salts or alkyl esters having 1 to 18 C atoms, e.g. methyl-, butyl-, or decyl stearate. It is also possible to use mixtures of these compounds as coating material. The customary ionogenic or non-ionogenic surfactants can be used as the emulsifier. In general an amount of 0.5 to 5 g, in particular 0.7 to 1.2 g, relative to 100 g of calcium carbonate particles, of these compounds is adequate.

The named compounds are added in emulsified form to the carbonate particles suspended in aqueous suspension, or CO<sub>2</sub> is conducted into the aqueous solution of Ca ions in the presence of the emulsified hydrophobic compounds. The water is subsequently removed.

The outer layers applied on one or both sides of the supporting layer can be both heat-sealable and cold-sealable. They can also be non-sealable layers that consist for example of the same polymer as the supporting layer. All these layers can be present on one or both sides. The two-sided outer layers can consist of the same or different materials. A heat-seal layer has a softening point that is at least 10°C lower than the softening point of the supporting layer. The seal layer consists preferably of an ethylene homopolymer (high-density polyethylene or low-density polyethylene), an LLDPE, a copolymer of propylene as the main component and ethylene, preferably with an ethylene proportion of 4 to 10% by weight (relative to the copolymer), a copolymer of propylene as the main component and 1-butene, preferably with a butene proportion of 10 to 15% by weight (relative to the copolymer), a terpolymer of propylene, ethylene, and an alpha-olefin having 4 to 10 C atoms, preferably one composed of 93.2 to 99.0% by weight of propylene, 0.5 to 1.9% by weight of ethylene, and 0.5 to 4.9% by weight of an alpha-olefin having 4 to 10 C atoms, or of a mixture of these polymers. The comonomers are essentially randomly distributed in the polymers. As particularly preferred seal layers, random alpha-olefin copolymers are used in which the ratio between the weight average  $M_w$  of the mole mass distribution to the number average  $M_n$  of the mole mass distribution is between 2.5 and 5.5.

The outer layer or the outer layers can also consist of a propylene homopolymer, whereby the melt index of the polymer used for the outer layer should have a value preferably higher by 0.5 to 1.0 g/10 min value than the melt index of the polymer used for the base layer (measured at 230°C and 2.16 kilogram-force load according to DIN 53 735).

Suitable cold-seal layers consist of polymers based on natural or synthetic rubber. Suitable external outer layers also consist of acrylates, as described in EP-A 0 214 790, EP-A 0 254 417, and CH-PS 632 777, or of vinylidene chloride copolymers, as named in EP-A 0 088 535. In a particular form of embodiment, the one outer layer consists of acrylate and the other outer layer of vinylidene chloride copolymer. If necessary, these compounds can be anchored with an adhesion-promoting layer on the underlying layer of polyolefin. Suitable primers are likewise listed in the named patents. While the cold-seal

layers are applied exclusively in a separate process step (off-line), the acrylates or vinylidene chloride copolymers can also be applied in-line before the longitudinal stretching. The in-line process is described e.g. in EP-A 0 214 790.

When the film is used as a packaging material, a lubricant is added to the outer layer(s) to improve its slip property with respect to satisfactory machine compatibility and with respect to frictionless filling of the bag with the goods to be packaged. It is advisable to use as the lubricant, a polydialkylsiloxane, preferably having 1 to 4 C atoms in the alkyl group, whereby polydimethylsiloxane is particularly preferred. The amount of the polydialkylsiloxane used in the outer layer or outer layers is 0.2 to 2.5% by weight, preferably 0.5 to 1.6% by weight, relative to the weight of the outer layers.

The total thickness of the film is 25 to 120 micrometers, preferably 30 to 60 micrometers, whereby the thickness of the outer layer(s) is 0.1 to 5 micrometers, preferably 0.6 to 2 micrometers respectively.

In order to improve certain properties of the film of the invention even further, both the base layer and the outer layer(s) can contain appropriate additives in a respectively effective amount; antistatics, e.g. N,N-bisethoxyalkylamine, and nucleating agents, e.g. sorbitols, are preferably named. The addition of processing stabilizers to the core layer has proved in particular to be favorable. These are compounds that are intended to prevent the thermal degradation of the polymers during the production of the film. These compounds include phosphite stabilizers and/or diphosphite stabilizers such as e.g. tris(2,4-di-*tert*-butylphenyl)phosphite and bis(2,4-di-*tert*-butylphenyl)pentaerythritol diphosphite in amounts of 0.05 to 0.3% by weight, relative to the core layer. Moreover, the addition of an optical brightener, e.g. bisbenzoxazoles, bis(styryl)biphenyls, triazine phenylcoumarins or benzotriazole phenylcoumarins, which is used in an amount of 100 to 500 ppm, in particular 150 to 250 ppm, relative to the core layer, has proved to be particularly advantageous. In particular to improve the formulating ability, 0.1 to 1% by weight, in particular 0.2 to 0.5% by weight, of an organic or inorganic antiblocking agent can be added to the outer layer or outer layers. Suitable antiblocking agents are for example organic polymers such as polyamide, polyester, polycarbonates, and the like, which are incompatible with the polymer in the outer layer and are present in the form of particles, or inorganic particles, in particular silicon dioxide.

The film can be produced by customary processing technologies for the production of multilayer films.

Thus the film can be produced by extrusion of the supporting layer or by co-extrusion of the molten polymer materials forming the individual layers through a flat film die, quenching and setting of the molten film, subsequent biaxial stretch orientation in the longitudinal and transverse direction, and final heat-fixing. For the biaxial stretching the film is heated and stretched first in the longitudinal direction and then in the transverse direction, or first in the transverse direction and then in the longitudinal direction. The longitudinal stretching takes place for example at 120 to 130°C in a ratio

of 5:1 to 7:1, and the subsequent transverse stretching at 160 to 170°C at a ratio of 8:1 to 10:1; the heat fixing takes place at 150 to 160°C for about 0.5 to 10 s. If required, the surfaces of the outer layers can undergo another surface treatment after the heat fixing, in order to endow the film with improved receptivity for the application of further coatings, and in order to improve its ability to be bonded or laminated, or printed or metallized.

The supporting layer is preferably produced using masterbatch technology, i.e. in accordance with the desired content of titanium dioxide- and calcium carbonate particles, polypropylene raw material, titanium dioxide masterbatch, and calcium carbonate masterbatch are added to an extruder to form the supporting layer. As is known, the term "masterbatch" is to be understood to mean a stock mixture, in particular a granular, non-dusting concentrate of a plastic raw material with large amounts of filler, which is used as an intermediate product in the preparation of the melt, thus as a pre-extrusion additive to granular material containing no fillers, in order to produce moldings containing certain amounts of filler therefrom. The masterbatch used according to the invention for adding  $\text{TiO}_2$  or  $\text{CaCO}_3$  excels in that it contains more than 30% by weight, preferably 40 to 75% by weight, of  $\text{TiO}_2$  particles or  $\text{CaCO}_3$  particles, relative to the total weight of polymers plus fillers, and that it is in the form of cylinder-shaped or pellet-shaped granular material. It is particularly advantageous to use a combined masterbatch containing 40 to 75% by weight, preferably 40 to 70% by weight, of  $\text{TiO}_2$ - and  $\text{CaCO}_3$  particles. Polyolefins, olefinic copolymers having 2 to 8 C atoms per olefin unit, or mixtures of these compounds are used as the carrier polymer for the masterbatch. The preferred carrier polymer is polypropylene, polyethylene, or a mixture of polypropylene and polyethylene. Abrasion phenomena, which occur during the extrusion processing of  $\text{TiO}_2$ -containing polyolefin, are clearly reduced by the preferred masterbatch process.

The supporting layer receives a large number of microcavities (vacuoles) through the longitudinal stretching. During the stretching procedure, the polymer matrix is torn open at the grain boundaries of the  $\text{TiO}_2$ - and  $\text{CaCO}_3$  particles, which leads to the formation of these cavities. Films of this type show a mother-of-pearl-like gloss and a lower density, which in the present case is 0.55 to 0.80 g/cm<sup>3</sup>, in particular 0.65 to 0.75 g/cm<sup>3</sup>.

The bulk density of the masterbatch is 700 to 1500 g/L, preferably 950 to 1250 g/L. The bulk density is determined using DIN 53 466. The ratio of the bulk density of the polypropylene raw material to the bulk density of the respective masterbatch is less than 0.80, preferably less than 0.65.

Owing to its advantageous properties, in particular its high and long-lasting degree of whiteness, the film of the invention is suitable as packaging material and also for technical purposes, e.g. as laminating film, label film, release film, or as supporting film for adhesive tape.

The invention is explained in more detail by the following Examples.

Example 1

A polypropylene film with heat-sealable surface layers present on both sides, composed of a random ethylene-propylene copolymer ( $C_2$  content 4.5% by weight), with the ratio of the weight average  $M_w$  (200 000 g/mol) to the number average  $M_n$  (50 000 g/mol) of the mole mass distribution being 4, is coextruded through a flat film die at about 270°C. The mixture (melt) of the core layer of the film consists of 91.6% by weight of polypropylene, 4.8% by weight of  $CaCO_3$  of an average particle size of 1 micrometer, and 3.6% by weight of  $TiO_2$  (rutile) with an average particle size of 0.2 micrometers, which has an inorganic coating of  $Al_2O_3$  (4 g/100 g of  $TiO_2$ ) as well as an organic coating of stearic acid (1 g/100 g of  $TiO_2$ ), whereby the mixture is formed from 82% by weight of polypropylene, 10% by weight of a masterbatch composed of 48 parts by weight of  $CaCO_3$  and 52 parts by weight of polypropylene, and 8% by weight of a masterbatch composed of 45 parts by weight of  $TiO_2$  and 55 parts by weight of polypropylene. The respective bulk density is 530 g/L for polypropylene, 720 g/L for the masterbatch with  $CaCO_3$ , and 750 g/L for the masterbatch with  $TiO_2$ .

The film is stretched as usual longitudinally and then transversely. The biaxially oriented film has a film thickness of 50 micrometers, whereby the seal layers are 0.8 micrometers thick respectively. The film density is 0.72 g/cm<sup>3</sup>. The surface gloss at a measuring angle of 20° is 25 scale divisions (measured according to DIN 67 530 with a Dr. Lange measuring instrument, Germany, Model RL 3).

Example 1a

In the same way as in Example 1, a polypropylene film with a surface layer present only on one side is produced from the same  $C_2/C_3$  copolymer and a 3 micrometer-thick surface layer of propylene homopolymer facing it. On this surface the film shows a gloss of 63 scale divisions at a measuring angle of 20° (DIN 67 530, Dr. Lange measuring instrument, Model RL 3).

Example 1b

Example 1a is repeated, whereby the 3 µm-thick polypropylene surface layer of Example 1a is additionally provided with an approximately 0.4 µm-thick layer of random  $C_2/C_3$  copolymer. The  $C_2/C_3$  copolymer has a  $C_2$  content of 4.5% by weight, and the ratio of the weight average  $M_w$  (200 000 g/mol) to the number average  $M_n$  of the mole mass distribution (50 000 g/mol) is 4. To improve the printability, this surface layer is subjected to a corona treatment. The surface tension is 40 mN/m. On this surface, in particular also at a low measuring angle, the film shows a high gloss (measuring angle



20°, gloss 72 scale divisions, DIN 67 530, Dr. Lange measuring instrument, Model RL 3), which is also clearly evident even after the film has been printed.

### Example 2

Example 1 is repeated, except that the  $\text{CaCO}_3$ - and  $\text{TiO}_2$  content is achieved by adding 12% by weight of a combined masterbatch of 30% by weight of  $\text{TiO}_2$ , 40% by weight of  $\text{CaCO}_3$ , and 30% by weight of polypropylene, to 88% by weight of polypropylene. The bulk density for the combined masterbatch is 1050 g/L.

### Example 3

Example 2 is repeated; the  $\text{TiO}_2$  particles are coated with  $\text{Al}_2\text{O}_3$  in the same way. Instead of stearic acid, polydimethylsiloxane (1.5 g/100 g  $\text{TiO}_2$ ) is used for the additional coating of the  $\text{TiO}_2$  particles.

### Comparative Example 1

Example 2 is repeated; the  $\text{TiO}_2$  particles are only coated with  $\text{Al}_2\text{O}_3$  (4 g/100 g  $\text{TiO}_2$ ).

### Comparative Example 2

Example 2 is repeated; the  $\text{TiO}_2$  particles are only coated with stearic acid (1 g/100 g  $\text{TiO}_2$ ).

### Comparative Example 3

Example 2 is repeated; the  $\text{TiO}_2$  particles remain uncoated.

The opacity, degree of whiteness, and yellowing tendency of the obtained film are shown in the following Table.

The opacity and the degree of whiteness are determined with the aid of an "ELREPHO" electric reflectance photometer made by the Zeiss company, Oberkochen (DE), standard light type C, 2° normal observer. The opacity is determined according to DIN 53 146. The degree of whiteness is defined as  $\text{WG} = \text{RY} + 3\text{RZ} - 3\text{RX}$ .

WG = degree of whiteness, RY, RZ, RX = corresponding reflectance factors when the Y, Z, and X color test filter is used. As the white standard, a pressed disk of barium sulfate (DIN 5033, Part 9) is used. A film sample (size DIN A4) is irradiated for several days in a device of the Suntest CPS type made by the Heraeus company, Hanau

(DE), irradiance  $765 \text{ W/m}^2$ , and is then compared with a non-irradiated sample with the naked eye.

	Opacity %	Degree of whiteness %	Yellowing tendency
Example 1/1a/1b	88	90	Very low
Example 2	92	93	Very low
Example 3	93	93	Very low
Comparative Example 1	76	78	Low
Comparative Example 2	82	76	Distinct
Comparative Example 3	76	74	Distinct

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INTERNATIONAL APPLICATION IN ACCORDANCE WITH THE AGREEMENT ON  
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